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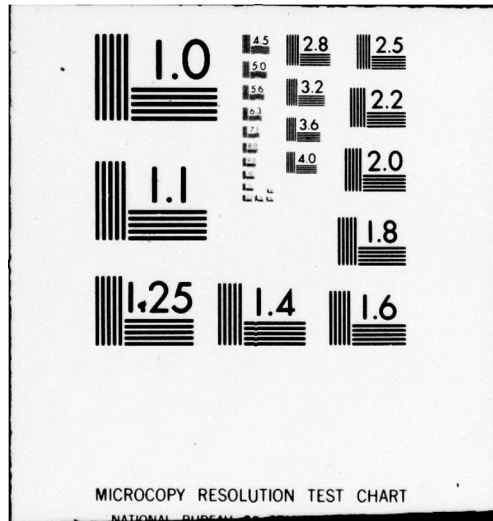
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EXTRACTION OF MINERAL ACIDS BY ALIPHATIC AMINES

By

Yu. G. Frolov, V. V. Sergiyevskiy



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EDITED TRANSLATION

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EXTRACTION OF MINERAL ACIDS BY ALIPHATIC AMINES

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Block	Italic	Transliteration	Block	Italic	Transliteration
А а	<i>А а</i>	A, a	Р р	<i>Р р</i>	R, r
Б б	<i>Б б</i>	B, b	С с	<i>С с</i>	S, s
В в	<i>В в</i>	V, v	Т т	<i>Т т</i>	T, t
Г г	<i>Г г</i>	G, g	У у	<i>У у</i>	U, u
Д д	<i>Д д</i>	D, d	Ф ф	<i>Ф ф</i>	F, f
Е е	<i>Е е</i>	Ye, ye; E, e*	Х х	<i>Х х</i>	Kh, kh
Ж ж	<i>Ж ж</i>	Zh, zh	Ц ц	<i>Ц ц</i>	Ts, ts
З з	<i>З з</i>	Z, z	Ч ч	<i>Ч ч</i>	Ch, ch
И и	<i>И и</i>	I, i	Ш ш	<i>Ш ш</i>	Sh, sh
Й й	<i>Й й</i>	Y, y	Щ щ	<i>Щ щ</i>	Shch, shch
К к	<i>К к</i>	K, k	Ъ ъ	<i>Ъ ъ</i>	"
Л л	<i>Л л</i>	L, l	Ы ы	<i>Ы ы</i>	Y, y
М м	<i>М м</i>	M, m	Ь ь	<i>Ь ь</i>	'
Н н	<i>Н н</i>	N, n	Э э	<i>Э э</i>	E, e
О о	<i>О о</i>	O, o	Ю ю	<i>Ю ю</i>	Yu, yu
П п	<i>П п</i>	P, p	Я я	<i>Я я</i>	Ya, ya

*ye initially, after vowels, and after ъ, ь; e elsewhere.
When written as ě in Russian, transliterate as yě or ě.

RUSSIAN AND ENGLISH TRIGONOMETRIC FUNCTIONS

Russian	English	Russian	English	Russian	English
sin	sin	sh	sinh	arc sh	sinh ⁻¹
cos	cos	ch	cosh	arc ch	cosh ⁻¹
tg	tan	th	tanh	arc th	tanh ⁻¹
ctg	cot	cth	coth	arc cth	coth ⁻¹
sec	sec	sch	sech	arc sch	sech ⁻¹
cosec	csc	csch	csch	arc csch	csch ⁻¹

Russian	English
rot	curl
lg	log

0554

EXTRACTION OF MINERAL ACIDS BY ALIPHATIC AMINES**Yu. G. Prolov, V. V. Sergiyevskiy**

Extraction by high-molecular aliphatic amines is widely used in the analytical and technological practice of a number of elements. As a rule the extractants of metal salts are not the amines, rather their salts, and the use of amines as extractants is preceded by saturation of the organic phase by the acid. It is for this reason that extraction of mineral acids by solutions of amines in organic solvents has been the subject of a great number of studies [1-4].

It should be mentioned that it is primarily the extraction of acids by tertiary amines which has been studied. Discussed in the present article are the laws of extracting various mineral acids by tertiary and secondary amines.

In this work we used "chemically pure" sulfuric and nitric acids. Perchloric acid of "analytical grade" was purified by distillation. Hydrochloric, hydrobromic, and hydriodic acids were prepared directly prior to use by dissolving gaseous HCl, HBr, and HI obtained by the standard method in distilled water.

The solvents were purified by simple distillation. Extraction was conducted in equal phase volumes. Agitation time was 1 h, temperature - $20 \pm 2^\circ\text{C}$.

The equilibrium concentration of acids in the aqueous phase was determined by titration using a standard alkali solution with a methyl red indicator. Concentration of acid in the organic phase was determined by titration with an aqueous solution of alkali in the presence of phenol phthalein.

In calculating the equilibrium constants the values of the activity coefficients of the acids introduced in [5] were used.

Discussion of Results

Extraction of acids by tri-n-octylamine. Analysis of the

results of various researchers [1-4] shows that during extraction of monobasic acids by solutions of tri-n-octylamine (TOA) an improvement is observed in extraction of acids in the series: $\text{HF} < \text{HCl} < \text{HBr} < \text{HNO}_3 < \text{HJ} < \text{HClO}_4$. Our results on extraction of acids by TOA (Table 1) confirmed this pattern. Since the equilibrium concentrations of acids in the aqueous phase does not exceed 0.05 M and the difference in activity coefficients for similar concentrations [5], then we can assume that the aqueous phase is not responsible for the difference in extractability of the acids observed here.

To explain the difference in extraction of acids the following factors were considered.

Since during the process of acid-base interaction the acid proton converts to a base molecule, then basically the different extractability of the acids can be explained by the energetic exchanges which occur during formation of the cation. The formation energy of the cation (ΔE_m) in the first approximation is equal to the difference in the affinity of the amine to the proton and the affinity of the anion to the proton, i.e.,

$$\Delta E_m = \Pi_m - \Pi_A^- \quad (1)$$

In the case of extraction by one amine $\Pi_m = \text{const}$, differences in the

formation energies of the cation are also caused by the change in the proton affinity of the ion, where, the greater the value Π_A^- , the lower is the extraction of acids. A comparison of Π_A^- [6]: ClO_4^- - 285 kcal/g-ion, J^- - 307 kcal/g-ion, Br^- - 315 kcal/g-ion, NO_3^- - 320 kcal/g-ion, Cl^- - 325 kcal/g-ion, F^- - 363 kcal/g-ion with an experimentally observed acid extraction series showed that the extractability of acids does indeed grow when their proton-donor properties are strengthened.

A differential in extraction of acids can also be anticipated as a result of the difference in association of salts, since, as we know, for TOA salts association intensifies in the series $\text{TOANCl} < \text{TOANBr} < \text{TOANClO}_4$, i.e., as the dipole moment of the salt increases [7].

Our attention is drawn to the slight difference in the extraction behavior of HClO_4 and HJ , as well as that of HBr and HNO_3 , despite the differentiating effect of the noted factors. Consequently, some processes and the energy changes which correspond to them act in the opposite direction. One of these is the formation of an intramolecular hydrogen bond between the cation and ion of the salt, whose existence in amine salts is established by means of various research methods: Nuclear magnetic resonance spectra [2, 8], IR-spectra [9, 10], conductometry [11], etc. We know that for salts

of TOA the intensification in the hydrogen bond occurs in the series: $F^- > Cl^- > NO_3^- > Br^- > I^- > ClO_4^-$, as one might assume on the basis of the proton-acceptor properties of the anions. It is obvious that intensification of the hydrogen bond, just as the change in energy of the electrostatic interaction during replacement of the anion [4], should lead to a leveling in the extractability of the acids.

Extraction of acids by amines of different classes during formation of normal salts. The results of Table 1 show that during the formation of sulfates of TOA and di-n-nonylamine (DNA) the extraction of sulfuric acid increases from TOA to DNA. The same thing occurs in the case of nitric acid. This is apparent from the constants established by measuring the pH, which for TOA equals 6.4, for DNA in benzene - 11.6.

Table 1. Stability constants of normal amine salts.

1 Соль	2 lg K в циклогексане	3 lg K в диэтилбензоле
TOAHClO ₄	6.42	6.67
TOAHJ	5.98	6.14
TOAHBr	3.96	4.00
TOAHNO ₃	4.07	4.64
TOAHCl	2.75	3.85
(TOAH) ₂ SO ₄	5.40	6.63
(DNAH) ₂ SO ₄	10.96	13.24

Key: 1) Salt; 2) lg K in cyclohexane; 3) lg K in diethylbenzene.

It is obvious that the difference in the equilibrium constants during extraction by TOA and DNA of 5-7 orders cannot be caused by the influence of any one factor. We can assume that replacement of the amine is to the greatest degree reflected in the formation energy of the cation, in the force of the intramolecular bond, and in the intermolecular association.

Since the value of affinity to the proton for TOA and DNA is unknown at the present time, to compare the formation energy of the cation we use values of 210.8 kcal/mole and 213.8 kcal/mole for trimethylamine and dimethylamine, respectively [12].

Differences in the formation energy of the cation of 3 and 6 kcal/mole correspond to an increase in the equilibrium constant for the secondary amine relative to the tertiary by 2 and 4 orders for mono- and dibasic acids, respectively.

In studying the energy of the intramolecular bond in salts it should be noted that the energy of the electrostatic interaction probably changes only slightly as a result of small differences in the dimensions of the amines. In explaining the results on solvation of amine salts it was earlier assumed that the presence in the nitrogen atom of various amines of the unreplaced proton intensifies the intramolecular hydrogen bond [13]. Increasing the energy of the hydrogen bond by 2-4 kcal/mole should lead to an increase in the equilibrium constant by 2-3 orders.

It has been experimentally established that another factor which changes greatly during the transition from salts of tertiary to salts of secondary amines is their intermolecular association. Studies by Allen on the scatter of light by solutions of amine salts showed that the mean number of particles in associates of di-n-decylamine sulfate (analog of the DNA sulfate) equals 40, while the TOA sulfate is virtually a monomer [14]. Based on these results and assuming that the number of particles in the associate of the DNA sulfate was constant, the activity coefficient was calculated. For 0.1 M of solution it proved to be equal to 0.056. Consequently, the increase in association of salts of secondary amines can explain an increase of only 10-20 times in the extraction constant.

From the above it is apparent that the increase in extraction of acids during the transition from tertiary to secondary amines is satisfactorily explained by the increased infinity of the amine to the proton and by the intensification of the intramolecular hydrogen bond and association of salts.

Extraction of second acid molecule by amines. We know that when the concentration of the acid in the aqueous phase is increased, the amount of acid which converts to the organic phase exceeds that necessary for formation of the normal salts of amines. It was assumed that the nonequivalent acid is distributed by the physical distribution mechanism [4] as a result of the attachment of the second acid molecule to the alkyl ammonium ion [15] or as the result of the formation of a hydrogen bond with anions of alkyl ammonium salts [10].

Since we demonstrated earlier [13], that molecules of protogenic solutions attach themselves to the ions of amine salts, the shift in extraction of nonequivalent acid in the range of more concentrated solutions of acids when such diluents are used is evidence in favor of this last proposal. From studies of various researchers [3, 4] it appears that the increase in extraction of the second molecule of

acid occurs in a series which is the reverse of that observed in extraction of the first molecule: $\text{HF} > \text{HCl} > \text{HBr} > \text{HClO}_4$. In this sequence the proton-acceptor properties of the anions decrease. This is evidence of the attachment of the second molecule by means of the hydrogen bond to the anion of the amine salt. Moreover, anions of the XHX^- type represent standard and stable formations in various nonaqueous solutions [16].

The results on extraction of hydrochloric acid by solutions of TOA and DNA in benzene (table 2) and on extraction of nitric acid by solutions of di-n-octylamine [17] and TOA [18] indicate that the stability constants of compounds of amine salts with the second acid molecule decrease from tertiary to the secondary amines. Extraction of the second nitric acid molecule by solutions of amines of different classes also decreases from tertiary to primary amines [19], i.e., we observe a pattern which is the opposite of the extraction of the first molecule of acid.

Table 2.

0,1 M TOA			0,1 M ДНА		
Равновесная кислотность, М		lg K	Равновесная кислотность, М		lg K
1) в водной фазе	2) в органич. фазе		1) в водной фазе	2) в органич. фазе	
3,90	0,121	2,36	3,90	0,1085	2,81
4,80	0,126	2,75	4,85	0,110	3,28
5,85	0,135	3,07	5,90	0,115	3,56
6,85	0,146	3,37	6,90	0,118	4,01
7,80	0,160	3,63	7,85	0,122	4,45
8,80	0,184	3,55	8,85	0,130	4,65
9,65	0,194	3,49	9,70	0,139	4,90

Key: 1) Equilibrium acid, M; 2) in aqueous phase; 3) in organic phase.

It is obvious that, just as in the case of attenuation of solvation by spirits of anions of amine salts in the series - tertiary, secondary, and below, the increase in the nonequivalent acid by salts of secondary amines is explained by intensification in them of the intramolecular hydrogen bond. Based on the donor-acceptor mechanism of the hydrogen bond we can assume that the formation of the intramolecular hydrogen bond in amine salt leads to a significant change in the distribution of the electron density of the anion. Intensification of the hydrogen bond in salts of secondary amines should lead to greater delocalization, as a result of which there develops a decrease in the electron-donor properties of the anions in relation to the conjugate hydrogen or donor-acceptor bond. Moreover, the presence of the unreplaced hydrogen atom in secondary and primary

amines greatly strengthens the association of their salts, apparently because of the formation of intermolecular hydrogen bonds. This also reduces the ability of amine salts to extract nonequivalent acids.

It should be mentioned that an analogous explanation can be given for the dependence of the extractability of metal salts by amine salts of different classes, which interact to form complexes in the organic phase. We observe the greatest possible drop in the symmetry of anions in the infrared spectra [20], which indicates their active participation in the complex-formation process. This confirms the belief of various researchers [21] that the anion complex of the metal is found within the internal sphere of the formed complex, while the alkyl ammonium cations are found on the outer coordination sphere, i.e., there is a donor-acceptor bond between the anion of the amine salt and the cation of the metal. Since in the transition from salts of quaternary ammonium to salts of primary amines we observe an intensification in the hydrogen bond, we can assume that the electron-donor properties in this same sequence decrease. Thus, a decrease in extractability should be observed in this series. An examination of many experimental data reveals that it is this dependence which we observe in the extraction of all salts of metals from nitric acid [22] and hydrochloric acid [23] media as well as most salts of metals from sulfuric acid media [24].

Effect of diluent on extraction of acids by tri-n-octylamine. To a great extent extraction equilibrium is determined by the nature of the diluent which is used. It has been established that the effect of the diluent is the result of the interaction of its molecules with both the molecules of the extractant and the molecules of the extracted complex [25] and that consideration of this interaction makes it possible to determine the nature of the dependence of the equilibrium constants on certain physicochemical characteristics of the diluents [26].

We know that the relationship between the equilibrium constant K_a , expressed in terms of absolute activities (standard state - pure components) and the equilibrium constant \dot{K}_a , expressed in terms of concentration activities (standard state - hypothetical solution with properties of infinitely diluted solution):

$$K_a = \dot{K}_a \dot{K}_{\gamma_0} \quad (2)$$

This equation is analogous to

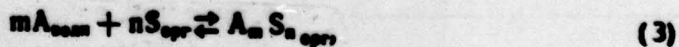
$$\gamma = \dot{\gamma} \gamma_0$$

where γ is the absolute coefficient of activity; $\dot{\gamma}$ - concentration coefficient of activity, and γ_0 - zero coefficient of activity (or absolute coefficient of activity of component when its concentration

moves toward zero).

Quantity K_{γ_0} represents the relationship between the zero activity coefficients of the components, and thus depends only on the medium. In the case of extraction it depends on the diluents which determine the phase.

Since the diluents which are used even partially dilute the water and are themselves diluted in it, then the value of constant K_{γ_0} is determined by the relationship of zero activity coefficients of all reaction components both in the organic and aqueous phases. Thus, equation (2) represents the most general equation which determines the effect of the organic diluent on extraction equilibrium. Only the assumption that the substances which determine the phase (water and diluent) are absolutely insoluble in one another makes it possible to determine the effect of the diluent by the relationship of zero activity coefficients of the components in the organic phase alone. Sometimes [26] it is erroneously assumed, for example, for the extraction reaction



which is the general thermodynamic equation which considers the effect of the organic diluent, not the equation

$$K_s = K_s \frac{T_{AS}^{\circ}}{T_s^{\circ} T_A^{\circ}}, \quad (4)$$

but the equation

$$K_s = K_s \frac{T_{AS}^{\circ}}{T_s^{\circ}} \text{ or } K_s = K_s \frac{T_s^{\circ}}{T_{AS}^{\circ}} \quad (5)$$

Quite frequently the fact of intersolubility is not considered in determining the activity coefficients of the substances by the distribution method. This sometimes leads (frequently for electrolytes) to great deviations from reality. This has been pointed out, for example, by the authors of [22].

Yet the assumption of the mutual insolubility of the phases simplifies determination of laws, particularly where solubility of the phases does not play a significant role.

Based on equation (5), using in the estimate quantities γ_{AS}° and γ_s° the formulas from the theory of regular solutions for determining the activity coefficients in binary solutions, Vdovenko, Kovaleva,

and Ryazanov [26] obtained a relationship which binds the extraction constant \dot{K}_2 with certain physicochemical peculiarities of the diluents:

$$\ln \dot{K}_2 = \ln K_2 + n \ln \gamma_S^0 - \ln \gamma_{AS}^0 - \ln K' - (n-1) RT \ln v_d - 2(nv_S \delta_S^2 - v_{AS} \delta_{AS}^2) \delta_d + (nv_S - v_{AS}) \left(\delta_d^2 - \frac{RT}{v_d} \right), \quad (6)$$

where $\ln K'$ does not depend on the nature of the diluent and equals

$$\ln K' = \ln K_2 + RT \ln \frac{v_S^0}{v_{AS}} + (n-1) RT + nv_S \delta_S^2 - v_{AS} \delta_{AS}^2; \quad (7)$$

v_S, v_{AS}, v_d are the molar volumes of the components in the solution;
 $\delta_S, \delta_{AS}, \delta_d$ - their solubility parameters.

The latter are determined by equation

$$\delta_i^2 = (\Delta H_i - RT)/v_i, \quad (8)$$

where ΔH_i is the molar heat of evaporation of the i -th component at temperature T ;

$$v_1 = M_1/d_1,$$

where M is the molecular weight; d - density.

From equation (6) it appears that within the framework of the theory of regular solutions the effect of the diluent is determined entirely by the values of the solubility parameters and the molar volumes of the diluents which are used.

We decided to test this equation on the findings by the influence of the diluent on the extraction of hydrochloric and sulfuric acids by solutions of tri-*n*-octylamine.

For a number of diluents with an approximately equal molar volume ($v_d \approx \text{const}$), for the amine-acid solution system (consequently, v_s , v_{AS} , δ_s and δ_{AS} - constant) equation (6) acquires the form of:

$$\ln K_e = A\delta_d^2 + B\delta_d + C \quad (10)$$

i.e., there should exist a parabolic dependence between the logarithm of the equilibrium constant and the solubility parameter of the diluent.

We know that for TOA $v_s = 433 \text{ cm}^3$, while for TOAHCl $v_{AS} = 424 \text{ cm}^3$

[27], i.e., in this case $\Delta = nv_s - v_{AS} > 0$, and the parabola should have a minimum at

$$t_s = \frac{v_{AS}^2 v_{AS} - nv_s^2 t_s}{nv_s - v_{AS}};$$

for dibasic acids the parabola should be steeper than for monobasic.

As we learn from Figs. 1 and 2, the shapes of the curves for extraction of hydrochloric and sulfuric acids by TOA correspond theoretically to those found, and, consequently, the effect of aprotic diluents on the extraction of acids by amines can be described within the framework of the theory of regular solutions with the interaction of both amine molecules and the molecules of amine salts considered.

Conclusion

1. It has been confirmed that extraction of monobasic acids by tri-n-octylamines during formation of normal salts increases in the series: $\text{HCl} < \text{HBr} \ll \text{HNO}_3 < \text{HJ} \ll \text{HClO}_4$.

It has been shown that differences in the proton affinity of

anions and the association of salts in the organic phase has a differentiating effect on the extractability of acids, while the formation of the intramolecular hydrogen bond has a leveling effect. It has also been established that under these conditions secondary amines are more effective extractants because of the strengthening of the intramolecular hydrogen bond, the association of the salts, and the affinity to the proton of the amines.

2. It has been established that during extraction of the second acid molecule salts of tertiary amines are more effective extractants than the corresponding salts of the secondary. This is explained by the decreased electron-donor properties of the anions as a result of the strengthening of the intramolecular hydrogen bond and their association.

3. Demonstrated here is the applicability of the equation introduced in [26] on the basis of the theory of regular solutions for description of the effect of a protonic diluents during extraction of acids by amines.

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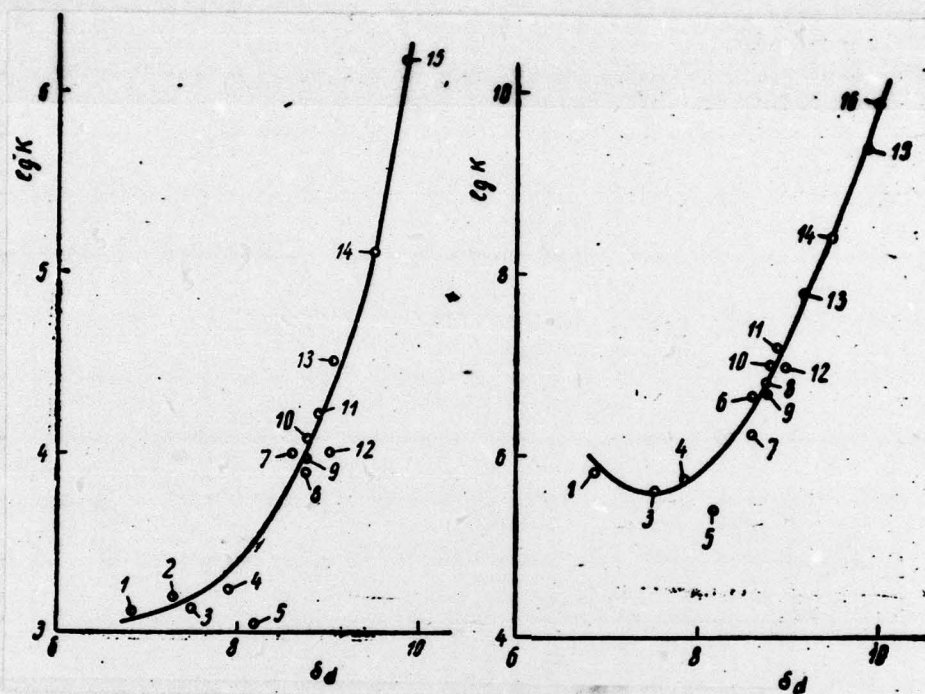


Fig. 1. Dependence of extraction constant of hydrochloric (a) and sulfuric (b) acids by tri-n-octylamine on solubility parameters of diluents: 1 - isooctane; 2 - hexane; 3 - octane; 4 - tridecane; 5 - cyclohexane; 6 - n-butyl benzene; 7 - carbon tetrachloride; 8 - i-propyl benzene; 9 - n-xylol; 10 - ethyl benzene; 11 - toluene; 12 - o-xylol; 13 - benzene; 14 - chlorobenzene; 15 - 1, 2-dichlorethane; 16 - nitrobenzene.

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C510 AIR MOBILITY R&D	1	E410 ADTC	1
LAB/FIO			
C513 PICATINNY ARSENAL	1	FTD	
C535 AVIATION SYS COMD	1	CCN	1
C591 FSTC	5	ASD/FTD/NIIS	3
C619 MIA REDSTONE	1	NIA/PHS	1
D008 NISC	1	NIIS	2
H300 USAICE (USAREUR)	1		
P005 DOE	1		
P050 CIA/CRB/ADD/SD	2		
NAVORDSTA (50L)	1		
NASA/NST-44	1		
AFIT/LD	1		
LLL/Code L-389	1		
NSA/1213/TDL	2		